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Magnetron Enhanced Reactive Ion Etching of Group-III Nitride Semiconductor Materials

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Magnetron enhanced reactive ion etch rates of GaN, AlN, and InN wide bandgap semiconductors were investigated as a function of cathode power, pressure, and flow rate in BCl_3 plasmas. Etch rates were obtained which were significantly higher than previously reported for dry etching of these materials. Surface analysis of etched samples revealed the presence of boron and chlorine residues. Etching produced a gallium surface deficiency in GaN extending 10 nm below the surface, and a preferential loss of nitrogen in InN. Etch rates were determined for the ternary alloys $In_{0.25}Ga_{0.75}N$ and $In_{0.75}Al_{0.25}N$ as a function of the addition of H_2, SF_6, and Ar to BCl_3. $In_{0.25}Ga_{0.75}N$ etch rates increased for additions up to 60% H_2, 20% SF_6, and 60% Ar concentrations in the gas mixtures, with higher additions producing a decrease in etch rates. For $In_{0.75}Al_{0.25}N$, etch rate increased slightly for Ar concentrations up to 40%, while H_2 and SF_6 additions reduced etch rates.

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INTRODUCTION

The III-V nitride semiconductor materials GaN, AlN, and InN, along with their associated ternary alloys $In_xGa_{1-x}N$ and $In_xAl_{1-x}N$, are wide bandgap semiconductors which have applications in areas such as short wavelength light emitters and optoelectronic devices, high voltage switches, piezoelectrics, and high speed/high temperature electronics. Device applications which have been reported include InGaN/AlGaN double heterostructure blue light emitting diodes (LED's) [1], high efficiency III-V nitride double heterostructure LED's [2], GaN p-n junction LED's [3,4], GaN photodetectors [5,6], GaN metal-semiconductor field effect transistors [7], and AlGaN/GaN high electron mobility transistors (HEMT's) [8]. For device fabrication these materials must be patternable into small structures, preferably by dry etching. Until now, the few reports on dry etching of these materials have indicated etch rates which are relatively low.

Reactive ion etching (RIE) of GaN in BCl_3 [9] and $SiCl_4$ [10] plasmas has resulted in fairly low etch rates, the highest rates reported being approximately 100 nm/min in BCl_3 at low pressures. Similarly, RIE of GaN in HBr, HBr/Ar, and HBr/H_2 gas mixtures has produced etch rates which were <70 nm/min [11]. Ar^+ ion milling has been performed on III-V nitride materials, with etch rates of only <60 nm/min reported for Ar^+ ion energies of 500 eV [12]. Etch rates as high as 180 nm/min have been reported for chemically assisted ion beam etching (CAIBE) of GaN at room temperature with a 500 eV Ar^+ ion beam directed onto the sample in a Cl_2 ambient, and by raising the temperature to 200°C an etch rate of 210 nm/min was attained [13].

Electron cyclotron resonance (ECR) discharges are known to produce high density plasmas at low pressures. ECR etching of III-V nitride materials has been reported in Cl_2/H_2 [14-16], $CH_4/H_2/Ar$ [14-17], CCl_2F_2/Ar [17], and BCl_3/Ar [17] gas mixtures, with the highest rate reported for ECR being 75 and 110 nm/min for GaN and AlN, respectively, in a $10Cl_2/15H_2$ plasma with 1000W microwave power [14]. ECR etching in $HI/H_2/Ar$ and $HBr/H_2/Ar$ plasmas has resulted in somewhat higher etch rates, reaching 110, 125, and 105 nm/min for GaN, AlN, and InN, respectively, in $10HI/10H_2/5Ar$ plasmas [18]. The dependence of etch rate on composition of the III-V ternary alloys $In_xGa_{1-x}N$ and $In_xAl_{1-x}N$ in both Cl_2/H_2 and CH_4/H_2 ECR plasmas has been reported [16] and showed the expected trend of higher etch rates in CH_4/H_2 and reduced rates in Cl_2/H_2 for increasing In mole fraction.

Magnetron reactive ion etching (MIE) is an attractive high density dry etching technique which is similar to RIE, but with the addition of a magnetic field to confine the plasma electrons in closed orbits close to the wafer. This minimizes electron loss to the chamber walls and increases ionization efficiency and reactive species generation, resulting in high etch rates. Compared to RIE, the magnetic field of MIE allows a higher density plasma discharge to be created which is able to sustain itself at relatively low

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cathode bias voltages and low pressures, resulting in low ion bombardment energies and therefore minimal etch-induced wafer damage. We have previously reported on MIE of GaAs and AlGaAs in BCl_3 [19,20], $SiCl_4$ [21,22], CCl_2F_2 [22-24], and $CH_4/H_2/Ar$ [25,26] plasmas. This technical report presents an investigation of MIE of GaN, AlN, and InN in BCl_3 plasmas, and MIE of the ternary alloys $In_xGa_{1-x}N$ and $In_xAl_{1-x}N$ in BCl_3/Ar, BCl_3/H_2, and BCl_3/SF_6 gas mixtures.

DESCRIPTION OF EXPERIMENT

The GaN (1 µm), AlN (1 µm), InN (0.4 µm), $In_{0.25}Ga_{0.75}N$ (0.4 µm), and $In_{0.75}Al_{0.25}N$ (0.3 µm) layers, with thicknesses shown in parentheses, were grown on semi-insulating (100) GaAs substrates using metal organic molecular beam epitaxy (MOMBE) [27,28]. The group III sources were triethylgallium, trimethylamine alane, and trimethylindium, with nitrogen as the group V source. Reaction of these sources took place in an ECR plasma source (Wavemat MPDR) operating at 200W power and 2.45 GHz. GaN and AlN layers were resistive, the InN strongly n-type ($10^{20}/cm^3$), and the ternary compounds n-type with carrier concentrations in the range 10^{18}-$10^{19}/cm^3$.

The grown layers were basically defective single crystals with a high density ($10^8/cm^2$) of stacking faults and threading dislocations. Extensive characterization of the films by x-ray diffraction transmission electron microscopy, photoluminescence, and secondary ion mass spectrometry showed them to be typical of the material currently used in commercially available light-emitting diodes [29]. For example, x-ray diffraction full-width-at-half-maximum was typically 400-900 arc-seconds as measured with a double crystal system. No measurable difference in dry etch rates was observed between samples grown under optimum conditions on either GaAs or Al_2O_3 substrates. This is expected under high density plasma conditions where generally the etching is limited by sputter desorption of the etch products. In contrast, material quality has a much larger effect on wet chemical etch rates where a strong correlation with x-ray peak widths was observed.

Etching experiments were performed in an MRC 710 magnetron ion etch system having a 50 cm diameter vacuum chamber evacuated by a turbomolecular pump to pressures in the 10^{-6}-10^{-7} Torr range. The water-cooled, 1000 cm^2 area cathode was driven by a 13.56 MHz power source. Bar magnets contained within the cathode body and situated on the chamber lid at the top of the reactor provide a uniform magnetic field (100 G) at the wafer location. Samples to be etched were fastened to the cathode surface with vacuum grease to provide good thermal contact. When etching with a mixture of gases, gas composition was adjusted by varying the flow rate through the individual flow meters. To prevent etching into the substrate, etch durations were chosen to produce etch depths which were less than film thicknesses. Etch depths were determined from Dektak profilometer measurements on etched samples patterned with AZ5214 photoresist, which was subsequently removed with an acetone rinse.

Etch rate was measured as a function of gas mixture composition, cathode power density, pressure, and flow rate. Etch rate studies as a function of these parameters can provide valuable information for understanding the mechanisms of the etching process. Surface analysis of control and etched samples was carried out using Auger electron spectroscopy (AES) measurements performed on a Perkin Elmer PHI 660 Auger microprobe with a 3kV electron beam to stimulate Auger transitions and a 4kV Ar^+ ion beam to sputter etch the sample. Secondary ion mass spectrometry (SIMS) measurements were performed on a Cameca 3F system. Etched sample surface morphology was examined by scanning electron microscopy (SEM) on patterned samples.

ETCH RATE MEASUREMENTS IN BCl_3 PLASMAS

Etch products for the group III elements in a BCl_3 plasma are most likely the chlorides of Ga, Al, and In, whereas nitrogen is probably removed in the form of pure N atoms or some complex with chlorine atoms such as NCl_3. In the results reported here for BCl_3 etching, etch rate magnitudes generally followed the order of volatility of the group III-chloride compounds [30,31], namely GaN>AlN>InN.

Magnetron reactive ion etch rates of GaN, AlN, and InN in BCl_3 plasmas are shown in Figure 1 as a function of cathode power density, along with the associated cathode bias voltages. The etch rates of these materials increase with power due to an increased generation of reactive chlorine species. In addition, the increased bias voltage at higher power densities leads to greater ion bombardment energies and enhanced sputtering of volatile species from wafer surfaces. The GaN etch rate of 300 nm/min for 0.5 W/cm^2 power density is significantly greater than the highest previously reported room temperature etch rate of 180 nm/min attained with chemically assisted ion beam etching (CAIBE) of GaN [13]. The AlN and InN etch rates shown in Figure 1 are appreciably higher than those reported in a $10BCl_3/5Ar$ ECR plasma at 250 V cathode bias [17]. The relatively low etch rates of InN are to be expected because of the low volatility of $InCl_x$ species. The highest cathode bias voltage of 100 V achieved at 0.5 W/cm^2 in Figure 1 is considerably lower than reported for RIE under similar conditions. For example, Lin, et al. [9] report a bias voltage of 231 V in a BCl_3 plasma which produced a GaN etch rate of 100 nm/min. The low MIE bias voltages should result in minimal etch-induced wafer damage. The data of Figure 1 indicate that further increases in etch rates can be attained with power densities greater than 0.5 W/cm^2.

Figure 2 shows etch rates as a function of BCl_3 flow rate. Gas residence time, which is the mean time a gas molecule remains in the process chamber before being pumped away, is inversely proportional to flow rate. For GaN the decrease in etch rate with increasing flow rate indicates that etching is taking place in the flow rate regime where gas residence times are relatively short and thus reactant gas is pumped away before reaction with the GaN wafer surface can take place. For AlN and InN, etch rate increase with increasing flow rate indicates that etch rate for these materials is limited by the
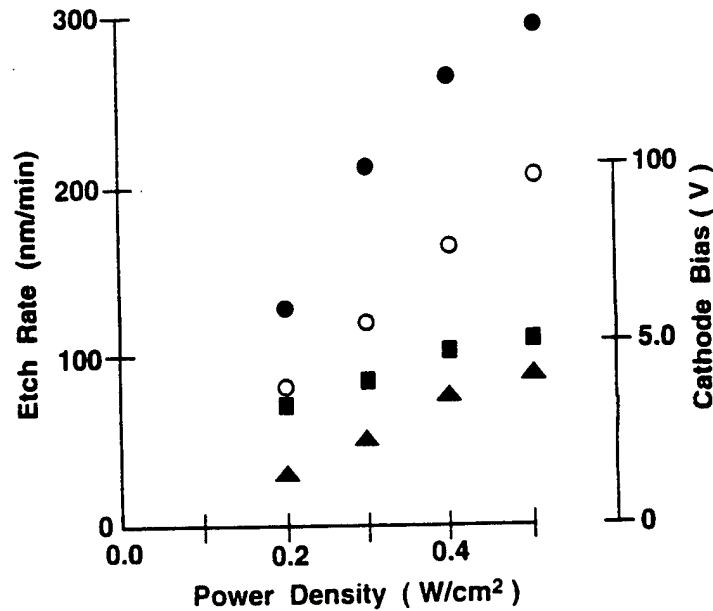
Figure 1. GaN (●), AlN (■), and InN (▲) etch rates as a function of cathode power density, with 0.5 sccm BCl_3 flow rate, 2 mTorr pressure. Also shown is cathode bias voltage (o) as a function of power density.
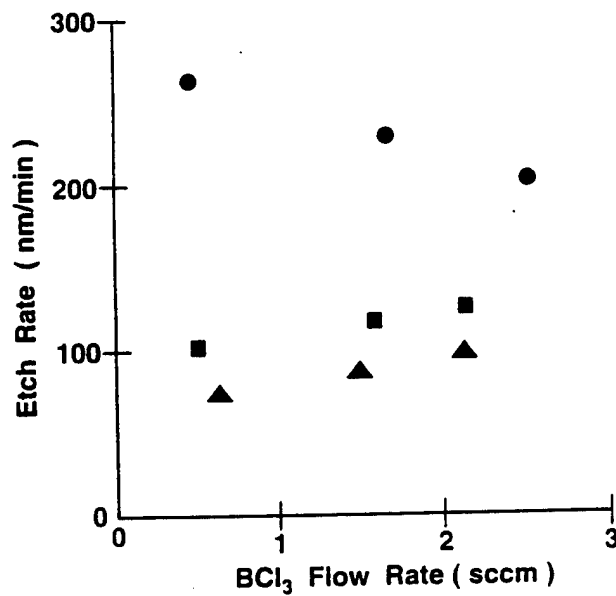

Figure 2. GaN (●), AlN (■), and InN (▲) etch rates as a function of BCl_3 flow rate, with 0.4 W/cm^2 cathode power density, 2 mTorr pressure.

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supply of etch gas over the given range of flow rates [32].

Figure 3 exhibits etch rates as a function of chamber pressure. Etch rate for GaN initially increased with pressure, reaching a maximum of 350 nm/min at about 7 mTorr, and then decreased with further pressure increase. The initial etch rate increase is indicative of a reactive-limited etch regime where a pressure increase allows more etch gas species to become available. AES measurements performed on a sample etched at the highest pressure (9 mTorr) showed the presence of a surface carbon deposit which is believed to be limiting the etch rate. Since this deposit was not observed on an unpatterned GaN sample etched under similar conditions, it appears that it is produced by the photoresist mask. A more appropriate etch mask should eliminate this etch rate-limiting effect.

Etch rate for AlN is essentially constant over the pressure range investigated, while that for InN increases slightly with pressure up to 7 mTorr and is saturated thereafter. These results suggest that BCl$_3$ etching is not limited by reactive gas supply for AlN under these conditions, but that at least up to 7 mTorr an increase in reactive chlorine species does produce faster etching of InN. Note that at pressures of 10-12 mTorr the etch rates of AlN and InN were essentially equal, suggesting that non-selective etching of InAlN/InN and InAlN/AlN heterostructures is possible under these conditions.
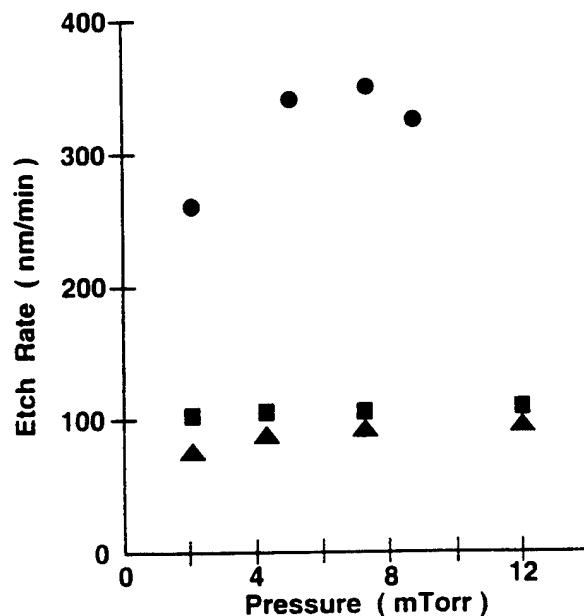

Figure 3. GaN (●), AlN (■), and InN (▲) etch rates as a function of pressure, with 0.5 sccm BCl$_3$ flow rate, 0.4 W/cm^2 cathode power density.

AUGER ELECTRON SPECTROSCOPY MEASUREMENTS

Figures 4(a) and 4(b) show AES surface spectra measured on a GaN control sample and an etched sample, respectively. The oxygen (O) and carbon (C) signals are due to air exposure. In addition to the constituent Ga and N signals, the etched sample exhibits a chlorine (Cl) residue with an estimated surface composition of <1 at.%. The Ga/N surface ratio decreased by about 25% upon etching, indicating a surface region Ga deficiency. AES depth profile measurements on the etched surface indicate that the Ga deficiency extends about 10 nm below the surface. Other workers have reported no change in surface stoichiometry of GaN ECR etched in Cl_2/H_2 plasmas [14] or ion milled with 500 eV Ar^+ ions [12].
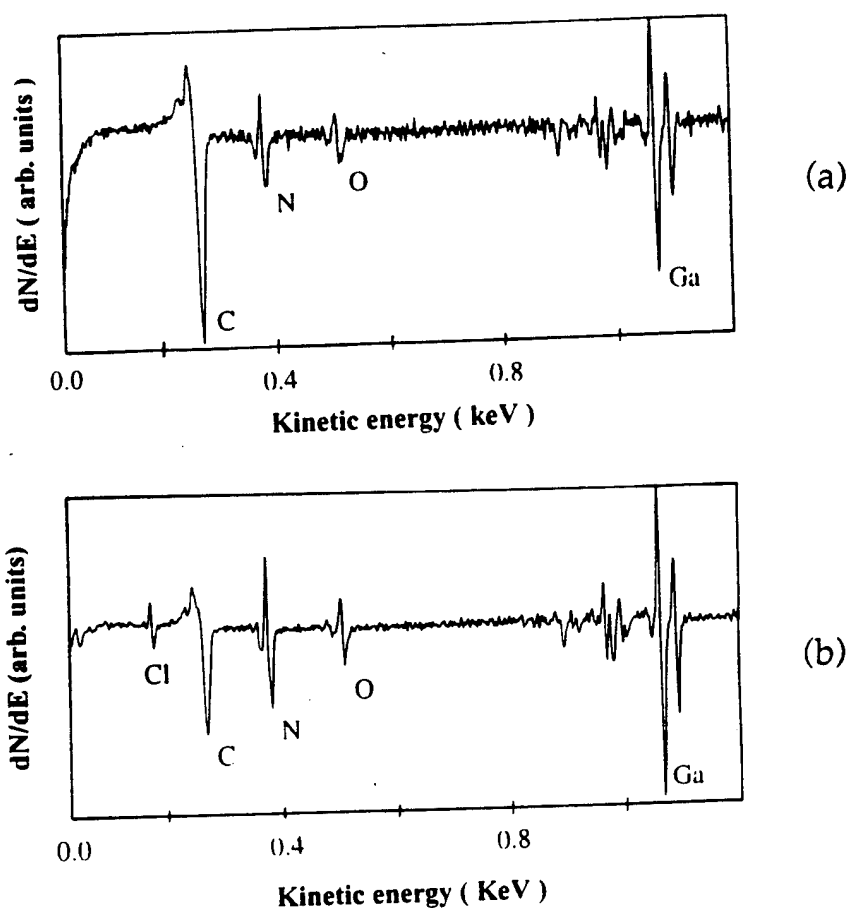

Figure 4. AES surface spectra of (a) GaN control sample and (b) GaN sample etched with 2 mTorr pressure, 0.4 W/cm^2 cathode power density, 0.5 sccm BCl_3 flow rate.

SCANNING ELECTRON MICROSCOPY MEASUREMENTS

Figure 5 shows a scanning electron micrograph of a GaN sample patterned with a Ni mask and etched to a depth of 0.6 µm. The etched surface retains the same features as the unetched, masked region. Figure 6 shows a scanning electron micrograph of a feature etched into AlN. The etching is smooth and anisotropic. Figure 7 is an SEM micrograph of a cross-sectional profile etched into InN. The sidewall defined by the mask edge exhibits a negative undercut characteristic which perhaps is indicative of a sputtering component required to remove the low volatility $InCl_x$ species. The as-etched AlN and InN surfaces had <1 at.% borine and chlorine residues as indicated by SIMS measurements, in addition to the usual oxygen and carbon signals due to air exposure. AES measurements on InN showed a preferential loss of N upon etching, similar to results reported for ECR etching in Cl_2/H_2 [14].
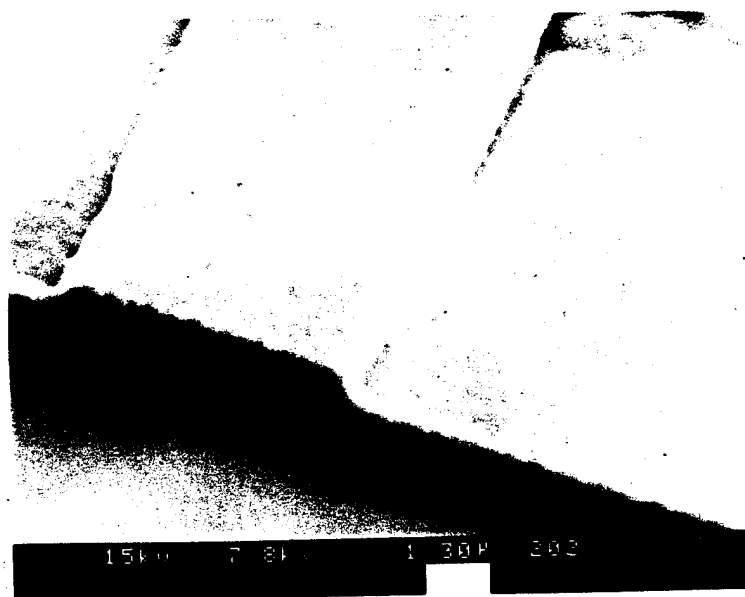

Figure 5. Scanning electron micrograph of GaN sample patterned with Ni mask and etched to a depth of 0.6 µm. Etching parameters were 0.5 sccm BCl_3 flow rate, 0.4 W/cm^2 cathode power density, 2 mTorr pressure, 2.5 min etch time.
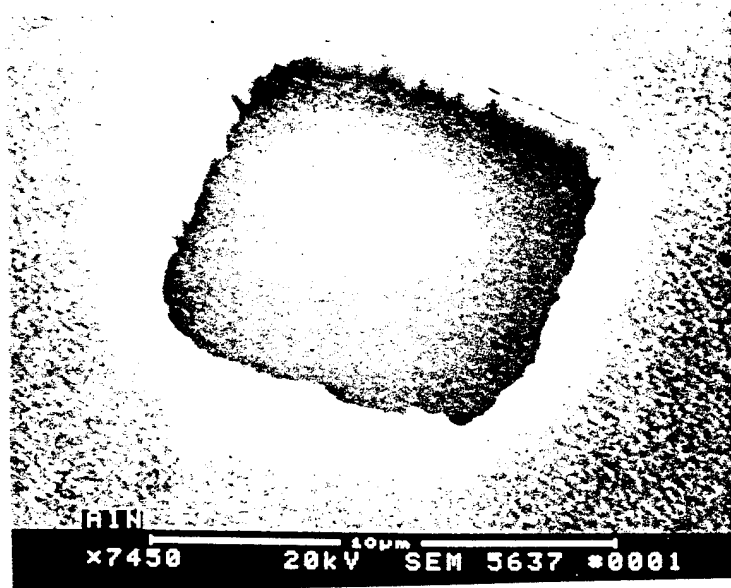
Figure 6. Scanning electron micrograph of AlN sample etched with 0.5 sccm BCl_3 flow rate, 0.4 W/cm^2 power density, 2 mTorr pressure.
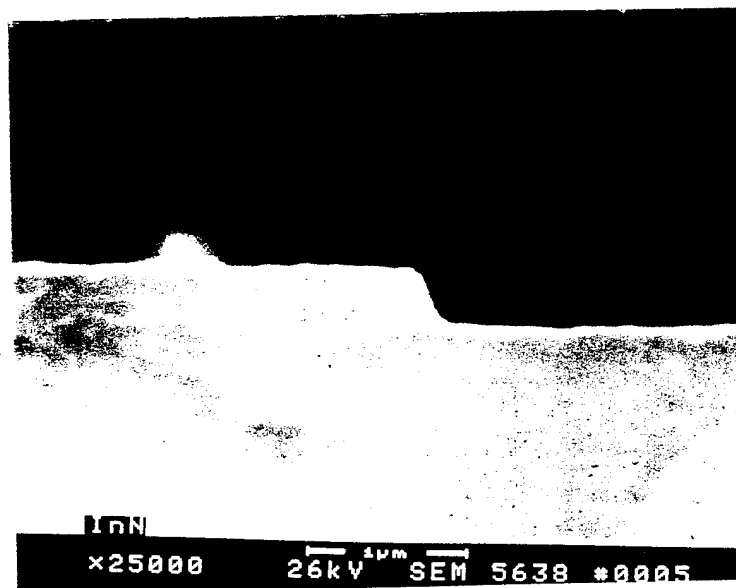

Figure 7. Scanning electron micrograph cross-sectional profile of InN sample etched with 0.5 sccm BCl_3 flow rate, 0.4 W/cm^2 power density, 2 mTorr pressure.

ETCH RATE MEASUREMENTS IN BCl_3 GAS MIXTURES

Pearton, et al. [16] have reported on the addition of H_2 and SF_6 to Cl_2 discharges for ECR etching of $In_{0.5}Ga_{0.5}N$ and $In_{0.5}Al_{0.5}N$ to enhance the removal of N in the form of

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volatile NH_3 and NF_3 etch products. The etch rates of both materials increased with the addition of up to 60% H_2 and 80% SF_6 in these discharges. However, Adesida, et al, [10] found that the addition of SF_6 to $SiCl_4$ produced essentially no change in the reactive ion etch rate of GaN.

Figure 8 shows $In_{0.25}Ga_{0.75}N$ and $In_{0.75}Al_{0.25}N$ MIE etch rates as a function of percent H_2 in H_2/BCl_3 mixtures. $In_{0.25}Ga_{0.75}N$ etch rate increased for H_2 additions up to 60% and then decreased for higher H_2 concentrations, similar to the H_2/Cl_2 etching results mentioned above. However, $In_{0.75}Al_{0.25}N$ etch rate was unchanged for H_2 additions up to 40%, above which the rate decreased. The eventual decrease in etch rates for low BCl_3 concentrations was due to less effective removal of the group III elements.
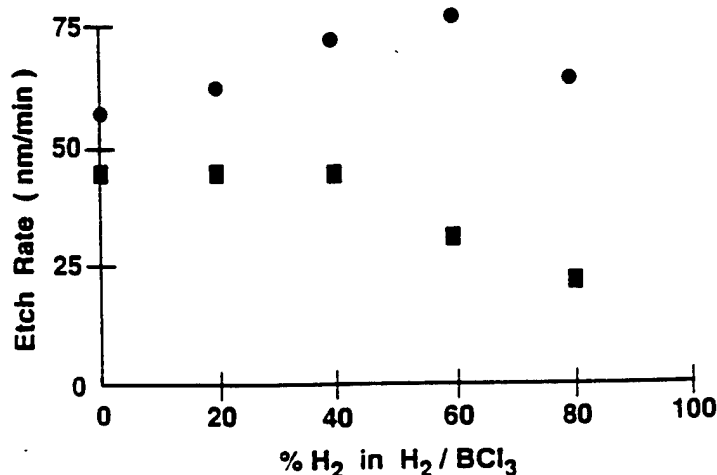

Figure 8. Etch rates of $In_{0.25}Ga_{0.75}N$ (●) and $In_{0.75}Al_{0.25}N$ (■) as a function of percent H_2 in H_2/BCl_3 mixtures, with 3 sccm total flow rate, 0.2 W/cm^2 power density, 2 mTorr pressure.

Figure 9 shows the effect of adding SF_6 to BCl_3 on the etch rates of these III-V nitride ternary compounds. The addition of 20% SF_6 produced a slight increase in $In_{0.25}Ga_{0.75}N$ etch rate, with further additions resulting in significantly lower rates. Adding any amount of SF_6 caused a reduction in $In_{0.75}Al_{0.25}N$ etch rate, probably due to formation of involatile AlF_3.

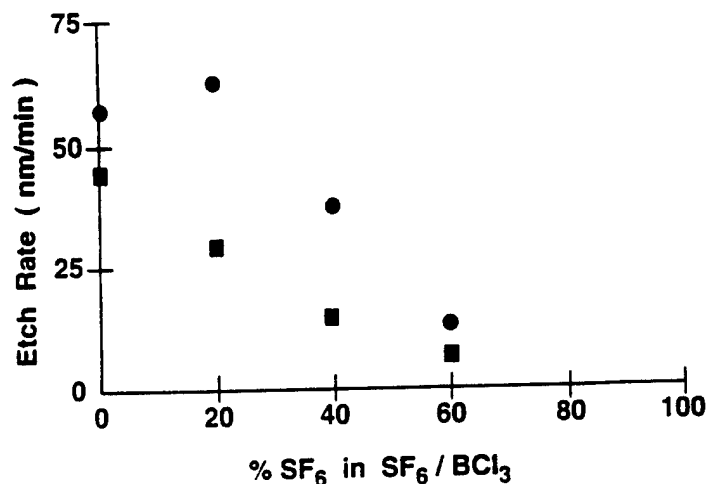Figure 9. Etch rates of $In_{0.25}Ga_{0.75}N$ (•) and $In_{0.75}Al_{0.25}N$ (■) as a function of percent SF_6 in SF_6/BCl_3 mixtures, with 3 sccm total flow rate, 0.2 W/cm^2 power density, 2 mTorr pressure.
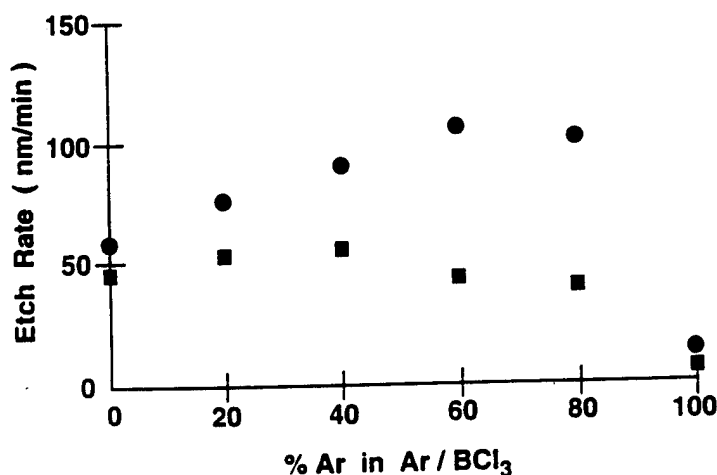

Figure 10. Etch rates of $In_{0.25}Ga_{0.75}N$ (•) and $In_{0.75}Al_{0.25}N$ (■) as a function of percent Ar in Ar/BCl_3 mixtures, with 3 sccm total flow rate, 0.2 W/cm^2 power density, 2 mTorr pressure.

The dependence of ternary alloy etch rates on Ar concentration in Ar/BCl_3 mixtures is exhibited in Figure 10. $In_{0.75}Al_{0.25}N$ shows a slight increase in etch rate for Ar concentrations up to 40%. However, $In_{0.25}Ga_{0.75}N$ etch rate increased significantly when Ar concentration reached 60%, producing an etch rate which was almost twice that for pure BCl_3. Since the etching process is due to a combination of chemical reaction and physical sputtering, etch rate increase at lower Ar concentrations was probably caused by increased sputter removal of etch products with the introduction of this heavy, chemically inert species. As Ar concentration was further increased, etch rates eventually decreased because physical sputtering began to dominate the etching

process, finally dropping to <10 nm/min for 100% Ar concentration.

SUMMARY

High etch rates of GaN, AlN, and InN were attained using the MIE technique with BCl$_3$ plasmas. Anisotropic etch characteristics were obtained along with smooth surfaces. Etch rates of In$_{0.25}$Ga$_{0.75}$N and In$_{0.75}$Al$_{0.25}$N were investigated as a function of H$_2$, SF$_6$, and Ar additions to BCl$_3$. MIE with BCl$_3$ chemistries is shown to be a suitable dry etching technique for low damage patterning of these wide bandgap materials.

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